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Comparison of Effects of Ethylene-Based and Propylene-Based Copolymer on Tailoring the Properties of Polypropylene

JINBO WU[®], BIN DANG, AND JUN HU[®], (Member, IEEE)

Department of Electrical Engineering, Tsinghua University, Beijing 100084, China

Corresponding author: Jun Hu (hjun@tsinghua.edu.cn)

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ABSTRACT Recently, polypropylene (PP) is considered as the potential recyclable HVDC cable insulation material. Ethylene-based copolymer (EBC) and propylene-based copolymer (PBC) were introduced into PP to tailor the properties of PP and make it more capable for HVDC cable insulation application. This paper focused on the effects of two additions on those propertied such as microstructure, thermal properties, electrical properties and space charge accumulation of PP. The results indicated that PBC was uniformly dispersed in the matrix of PP without adhesion, and its particle size did not increase with the increase of its content in the matrix. With the increase of PBC, the glass transition peak of polypropylene decreased slowly, indicating a better performance on flexibility at low temperature. With the increase of content of copolymer, the DC volume resistivity of the composite material decreased gradually, but samples with PBC had higher resistivity than samples with EBC. In the term of space charge, the addition of PBC could help suppressing the accumulation of space charge better. To sum up, PP/PBC composite material could be an option for recyclable DC cable insulation material.

INDEX TERMS Ethylene-based copolymer, propylene-based copolymer, polypropylene, DC cable, space charge.

I. INTRODUCTION

Compared with AC transmission system, HVDC transmission technology has the advantages of unrestricted transmission distance, low loss, and interconnection between power grids with different frequencies, providing an effective solution for current and future power grid [1]-[3]. DC transmission system includes transmission channels and converter stations at both ends, between which the most commonly used transmission channels are overhead transmission lines. However, due to the shortage of land resources, the power transmission channels are facing severe challenges [4]-[6]. On the other hand, offshore wind power has become an important strategic emerging industry, nor can this power be sent over overhead transmission lines. The development of DC cable is a feasible solution to the transmission channel problems of HVDC and offshore wind power. Compared with overhead transmission lines, cable transmission has the advantages of low failure rate, no environmental impact and no environmental damage [7], [8].

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According to the manufacturing process, DC cables can be divided into wrapped DC cables and extruded DC cables, and the widely used insulating material in extruded DC cable system is cross-linked polyethylene (XLPE). Nordic Chemical industry is in the leading position in insulating material, and its LE4258 DCE can meet the requirements of insulation materials for ± 525 kV high-voltage DC cable [4]. Japanese researchers put a lot of effort into the research of insulation materials for DC cables as well. They produced DC cables by adding nanoparticles to AC cables and achieved good results [9]. It was proved that the XLPE performed better in mechanical properties at high temperatures, but its long-term operating temperature was only 70 °C, which could not meet the demand of large capacity DC transmission [10], [11]. On the other hand, it is difficult to recycle again by melting due to its special crosslinking network, and can only be disposed by incineration or landfill, which not only wastes resources, but also causes great damage to the environment.

As common chemical products with abundant sources and relatively cheap price, PP does not need crosslinking in cable extrusion and can be recycled after use, which can be

TABLE 1. The ratio of blending materials.

Group	Mess fraction (%)			
	PP	PBC	EBC	
PP	100	-	-	
EBC10	90	-	10	
EBC20	80	-	20	
EBC30	70	-	30	
EBC40	60	-	40	
PBC10	90	10	-	
PBC20	80	20	-	
PBC30	70	30	-	
PBC40	60	40	-	

considered as the potential recyclable HVDC cable insulation material. However, the glass transition temperature of polypropylene is around room temperature, so its elastic modulus can hardly meet the requirement of cable mechanical properties [12]–[14]. To solve it, ethylene-based copolymer (EBC) and propylene-based copolymer (PBC) were introduced into PP to tailor the properties of PP [15], [16]. Meanwhile, the electrical properties of the composites may be greatly affected by the fact that DC cables are usually operating above 50°C and the copolymer is molten at high temperatures, which poses a challenge to the electrical properties of composites at high temperatures.

In the present work, the preparation method of PP/copolymer composites was studied, and the effects of EBC and PBC on the thermal properties, mechanical properties, dynamic thermal mechanical properties and electrical properties of polypropylene were compared. The work provides an insight into tailoring the properties of PP and the reference for the development of recyclable DC cable insulation material.

II. MATERIAL PREPARATION AND TESTING

A. THE PREPARATION OF COMPOSITE MATERIAL

The PP (PP5722E1) used in this work is homogeneous polypropylene from ExxonMobil, and its melt flow rate (MFR) is 4g/10min (2.16kg @190°C). The ethylene-based copolymer (POE8452) is an ethylene-octane copolymer with a melt flow rate of 3g/10min (2.16kg @190°C) from Dow Chemical, USA. Its glass transition temperature is -51° C and its melting point is 66 °C. The propylene-based copolymer (SN0285) is from Mitsui Chemical, Japan, with a melt flow rate of 1.4g/10min and a softening temperature of 116°C. As the reference group, cross-linked polyethylene (LE4253DC) is from Nordics. Its melt flow rate is 2g/10min and the density is 0.922g/cm³.

The polypropylene and copolymer were firstly vacuumdried for 12 h at 100 °C, and then melted and blended with a mixer at 175 °C. The ratio of blending materials is shown in Table 1.

To prepare the PP and its composite material samples, the blending materials were preheated at 200 °C for 10min to make the material fully melt. Then they were gradually pressurized to 15 MPa and kept for 10min. Finally, the samples were water-cooled at 15MPa. The preparation of XLPE was as follows. The particles of XLPE were preheated at 140 °C for 10min, then gradually pressurized to 15 MPa after melting and kept at this pressure for 10min. After that, the samples were cross-linked at 180°C for 15min. Finally, the samples were water-cooled at 15MPa, and then kept for 24h in a vacuum oven at 70°C.

The samples were short circuited in a 100°C vacuum oven for 10h to eliminate residual internal charge before electrical tests.

B. MORPHOLOGY OBSERVATION

Hitachi 8010 scanning electron microscope was used to observe the microscopic morphology of the samples. Firstly, the samples were brittle broken in liquid nitrogen to maintain the microscopic morphology of the samples. The microscopic interfaces of the samples were observed at 1kV voltage. The samples were placed in nheptane at 90°C for 12h and then the surfaces of the samples were sprayed with platinum. The phase structures of the sample were observed at 20kV voltage.

C. THERMAL CHARACTERISTIC MEASUREMENT

Q2000 differential scanning calorimeter from TA was used to measure the melting point, crystallinity and crystallization temperature of the samples. Samples of 5 ± 0.1 mg were tested under the protection of nitrogen. The samples were kept at 20°C for 5min, and were heated to 200°C in 5min. Finally they were cooled to 20°C at 10°C/min.

D. MECHANICAL PROPERTY TEST

The tensile tests were performed on the dumbbell-shaped samples at the rate of 50mm/min, until the samples were pulled off. The stress-strain curves were obtained in the tensile process. Five samples were repeatedly tested for each sample to obtain the yield modulus, tensile modulus and elongation at break.

E. DYNAMIC THERMAL-MECHANICAL PERFORMANCE TEST

Q800 dynamic thermal-mechanical analyser from TA was used to test the mechanical performance of samples at different temperatures. The sample size was 10mm*5mm*200 nm. The samples were tested under sinusoidal force with a frequency of 50Hz and a strain of 0.005. The experimental temperature was from -100 to 100°C and the heating rate was 3°C/min.

F. RESISTIVITY MEASUREMENT

The leakage current curves of samples at different temperatures and different electric fields were measured.

G. SPACE CHARGE MEASUREMENT

A space charge measurement system was designed and Pulsed Electro-Acoustic method (PEA) was adopted to

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measure the space charge distribution of samples. The highspeed narrow pulse vibrated the charge inside the insulation material and generated sound signal, which was then converted into the voltage signal by the sensor of the lower electrode. The voltage signal was amplified and transmitted to the oscilloscope for storage.

III. RESULTS AND DISCUSSION

A. COMPARISON OF MICROSTRUCTURE

The phase structure of PP/copolymer includes the particle size of the copolymer and the distribution of the copolymer in polypropylene.



FIGURE 1. The dispersion of EBC in PP (a) EBC10 (b) EBC20 (c) EBC30 (d) EBC40.

The dispersion diagrams of EBC with different contents were shown in figure 1. There were two-phase structures in PP/EBC, where the black holes represented the copolymers etched by n-heptane. The figure indicated that the EBC was uniformly distributed in the matrix and presented a typical island structure, in which polypropylene was the continuous phase and the copolymer was the dispersed phase. When the content of EBC was 10%, the size of the dispersed phase was less than 2 um, indicating a good compatibility between PP and EBC. With the increase of copolymer content, the particle size of EBC increased gradually. When the content of EBC was less than or equal to 30%, the copolymer was a sphere in the matrix, and no interconnection occurred between the spheres. However, when the content reached 40%, the copolymers began to interconnect with each other and took on an irregular shape.

In comparison, the dispersion of PBC with different contents in polypropylene was shown in figure 2. The PBC was uniformly dispersed in the matrix without adhesion, and its particle size was about 200nm. Unlike EBC, the particle size of PBC did not increase with the increase of its content in the matrix. It could be seen that PBC had excellent compatibility with matrix, which laid a good foundation for its thermal, mechanical and insulating properties.

Meanwhile, the dielectric loss tangent in the dynamic thermal-mechanical performance test could reflect the



FIGURE 2. The dispersion of PBC in PP (a) PBC10 (b) PBC20 (c) PBC30 (d) PBC40.

molecular motion state and indicated whether there was phase separation or not [17]. The dielectric loss tangent of PP/copolymer was shown in figure 3, with PP as the reference group.



FIGURE 3. The comparison of dielectric loss tangent of PP/EBC and PP/PBC. (a) EBC (b) PBC.

As could be seen from figure 3(a), there were two significant relaxation peaks in the curve. The relaxation peak 1 $(-30^{\circ}C)$ was the glass transition temperature of EBC, and the relaxation peak 2 $(10^{\circ}C)$ was the glass transition temperature of PP, indicating that there was serious phase separation between PP and EBC. However, there was only one relaxation peak in the curve of PP/PBC in figure 3(b), indicating the homogeneous structure of PBC and polypropylene.

Mechanical loss peaks of PP and PP/copolymer were summarized in Table 2. With the increase of PBC, the glass transition peak of polypropylene decreased slowly. When the content of PBC reached 40%, the glass transition temperature of the composite decreased by 9.4°C. The lower glass transition temperature indicated that the composite material had great flexibility at low temperature.

B. COMPARISON OF THERMAL PROPERTIES

In order to increase the transmission capacity, it is necessary for the cables to operate at higher temperatures. It is reported that adding copolymer contributes to improving the mechanical properties of polypropylene at room temperature, but may affect its melting point [18]–[21].

The melting curves of PP/copolymer were shown in figure 4, in which XLPE was the reference group. The heat

TABLE 2. Mechanical loss peaks of PP and PP/Copolymer.

Group	Peak 1 (°C)	Peak 2 (°C)
РР	18.2	-
EBC10	15.7	-28.7
EBC20	14.7	-35.0
EBC30	14.8	-35.2
EBC40	12.9	-32.5
PBC10	12.3	-
PBC20	8.3	-
PBC30	4.1	-
PBC40	3.5	-



FIGURE 4. The melting curves of PP/EBC (a) EBC (b) PBC.

absorption peak in the melting curve can reflect the melting point. As could be seen from the figure, the melting points of both EBC and PBC were similar to that of PP, at about 160°C, which was about 40°C higher than that of XLPE. The melting point of PP/copolymer decreased slightly with the increase of copolymer content. On the other hand, the melting point of the semi-crystalline polymer was mainly determined by the thickness of the sheet [22], [23], which indicated that the addition of copolymer did not cause changes in the thickness of polypropylene sheet.



FIGURE 5. The non-isothermal crystallization curves of PP/EBC (a) EBC (b) PBC.

The non-isothermal crystallization curves of PP/copolymer were shown in figure 5. The addition of copolymer caused a slight decrease of the crystallization temperature. As could be seen from figure 5(a), EBC30 and EBC40 showed significant crystallization peaks at 50°C and 110°C, indicating that EBC was separated from polypropylene in the samples with high content of EBC. That was consistent with the observed results in SEM above.

TABLE 3. Thermal properties of PP and PP/Copolymer.

Group	Melting temperature(°C)	Crystalli nity (%)	Crystallization temperature (°C)	Crystallization enthalpy (J/g)
XLPE	106.2	39.2	91.3	92.5
PP	163.5	50.2	110.0	105.4
EBC10	164.9	43.4	111.0	94.2
EBC20	164.1	36.3	110.3	85.0
EBC30	163.0	29.8	110.2	74.6
EBC40	163.4	25.9	109.9	64.1
PBC10	163.6	47.1	109.3	98.4
PBC20	163.9	42.5	108.5	88.3
PBC30	163.9	37.1	108.0	77.1
PBC40	163.9	31.7	107.1	66.2

The thermal properties of samples were summarized in Table 3, including melting temperature, crystallinity, crystallization temperature and crystallization enthalpy. It was indicated that the crystallinity of PP reached 50.2%, 11.0% higher than that of XLPE. In addition, the crystallinity of PP decreased with the increase of the content of both copolymers. In the non-isothermal crystallization process, with the increase of EBC, the crystallization temperature of polypropylene rose at first and then slowly decreased.

C. COMPARISON OF MECHANICAL PROPERTIES

The high elastic modulus of PP at room temperature is the biggest challenge to use as the insulating material. Conventional XLPE has a tensile modulus of about 200MPa at room temperature, but the tensile modulus of PP is about 1000MPa at room temperature. In this work, the mechanical properties of polypropylene were improved by adding PBC, and the test results were compared with traditional XLPE and PP/PBC to optimize the addition content.



FIGURE 6. The stress-strain curves of PP/EBC and PP/PBC (a) EBC (b) PBC.

The stress-strain curves of samples were shown in figure 6. With the increase of EBC, the elongation at break of PP/EBC first increased and then decreased. When the content of EBC was 30%, the elongation at break of the composite reached the maximum. With the further increase of EBC, the elongation at break of the samples decreased, which was due to the interconnection of EBC in the matrix. Meanwhile, the elongation at break of PP/PBC was higher than that of PP.

The mechanical properties of XLPE, PP, PP/EBC and PP/PBC were summarized in Table 4. With the increase of copolymer content, the yield modulus and tensile modulus

Group	Yield modulus (MPa)	Tensile modulus (MPa)	Elongation at break (%)
XLPE	9.1±0.5	134±19	1028±103
PP	31.0±2.2	656 ± 59.1	1313±127
EBC10	30.6±1.6	703 ± 38.8	1382±152
EBC20	24.1±2.1	592±28.2	1344±150
EBC30	20.7±2.3	446±34.8	1452±116
EBC40	15.6±1.7	327±21.6	912±547
PBC10	26.8±2.5	580±65.4	1386±165
PBC20	22.4±3.5	446±87.8	1416±128
PBC30	16.1±1.9	276±26.0	1660 ± 76
PBC40	14.7 ± 1.1	243±32.2	1489±113

TABLE 4. Mechanical properties of PP and PP/Copolymer.

of PP decreased gradually. When the content of PBC was 40%, the tensile modulus of the sample was 243MPa, which was close to that of XLPE. Therefore, in terms of mechanical properties at room temperature, these samples can meet the requirement of cable insulation materials.



FIGURE 7. The energy storage modulus of PP/EBC and PP/PBC at various temperature. (a) EBC (b) PBC.

The energy storage modulus of various samples were shown in figure 7. With the increase of copolymer content, the energy storage modulus of composite material gradually decreased. At room temperature, the energy storage modulus of EBC40 and PBC40 were similar to that of XLPE, which was consistent with the results of the stress-strain curve of the material. At 100°C, the energy storage modulus of EBC40 and PBC40 was higher than that of XLPE at 70°C, which indicated that the composite material was sufficiently rigid at high temperature.

D. COMPARISON OF ELECTRICAL PROPERTIES

The volume resistivity of the insulating material determines the loss rate of the HVDC cable transmission system. Lower resistivity will generate more heat inside the material, increasing the temperature of the insulating material, and thus reducing its service life.

The leakage current curses of various samples were shown in figure 8. The electric field and the temperature were chosen to be the regular operating condition. As could be seen from figure 9, the resistivity of PP/PBC was obviously better than that of PP/EBC. When the content of PBC reached 40%, the resistivity of PP/PBC was still higher than that of samples with just 10% of EBC.











FIGURE 10. The space charge distribution of various samples (a) EBC40 (30°C) (b) PBC40 (30°C) (c) EBC40 (50°C) (d) PBC40 (50°C).

E. COMPARISON OF ELECTRICAL PROPERTIES

The space charge diagrams of PP/EBC and PP/PBC at different temperatures at a DC field of 40kV/mm with a polarization of 600s were shown in figure 10. It was indicated that at room temperature, there was very little charge injection in PP/copolymer. When the temperature was 50°C, two peaks occurred in PP/EBC samples and increased gradually with the polarization time. The induced charge at the cathode increased and the induced charge at the anode decreased, indicating that the negative charge accumulated in the material was more than the positive one. Meanwhile, the space charge distribution in PP/PBC samples was still excellent at 50°C. It can also be proved from the distribution of electrical filed in figure 11. PP/PBC performed better at 50°C.



FIGURE 11. The electrical field distribution of various samples (a) EBC40. (30° C) (b) PBC40 (30° C) (c) EBC40 (50° C) (d) PBC40 (50° C).



FIGURE 12. The space charge distribution of various samples in depolarization procession. (a) EBC40 (30°C) (b) PBC40 (30°C) (c) EBC40 (50°C) (d) PBC40 (50°C).

The space charge diagrams of PP/EBC and PP/PBC in the depolarization procession were shown in figure 12. The accumulated charge rapidly decreased at the beginning of depolarization, followed by a gradual decrease in the decay rate. It was obvious that a large amount of charge accumulated near both the cathode and the anode in PP/EBC samples at 50°C. The space charge density near the cathode reached 20C/m³, while the one near the anode reached 30C/m³.

IV. CONCLUSION

In this work, composited material of PP/PBC and PP/EBC were prepared, and the effects of PBC and EBC on the thermal properties, mechanical properties, dynamic thermal

mechanical properties and resistivity of PP were compared. The relationship between microstructure and properties of composites was investigated. Based on the experimental result in the present work, the following conclusions were drawn.

Compared with EBC, the PBC was uniformly dispersed in the matrix of PP without adhesion, and its particle size did not increase with the increase of its content in the matrix. With the increase of PBC, the glass transition peak of polypropylene decreased slowly, indicating a better performance on flexibility at low temperature. With the increase of content of copolymer, the DC volume resistivity of the composite material decreased gradually, but samples with PBC had higher resistivity than samples with EBC. In the term of space charge, the addition of PBC could help suppressing the accumulation of space charge better. To sum up, PBC performed better than EBC on tailoring the thermal properties, mechanical properties, dynamic thermal mechanical properties and resistivity of PP, thus PP/PBC composite material could be an option for recyclable DC cable insulation material.

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JINBO WU was born in Shanxi, China, in 1993. He received the B.Sc. degree in electrical engineering from Tsinghua University, Beijing, China, in 2015, where he is currently pursuing the Ph.D. degree with the Department of Electrical Engineering. His research interests include ZnO varistor and other nonlinear materials.



BIN DANG was born in Shaanxi, China, in 1989. He received the B.Sc. degree from Sichuan University, China, in 2013, and the Ph.D. degree in electrical engineering from Tsinghua University, Beijing, China, in 2018. His research fields include nanodielectrics, space charge phenomenon in insulation materials.



JUN HU (Member, IEEE) was born in Ningbo, Zhejiang, China, in 1976. He received the B.Sc., M.Sc., and Ph.D. degrees in electrical engineering from the Department of Electrical Engineering, Tsinghua University, Beijing, China, in July 1998, July 2000, and July 2008, respectively.

He is currently an Associate Professor with the Department of Electrical Engineering. His research interests include overvoltage analysis in power systems, dielectric materials, and surge arrester technology.

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